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Final Report

by


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## Macrocycles Containing Tin

The project involved the development of methods for the synthesis, purification and characterization of pre-organized macromolecular hosts containing several Lewis acidic tin atoms, and investigations of the potential for such compounds to bind anions and basic donors selectively. The rationale for the study was that macrocyclic species containing Lewis acidic sites should bind anions and donors within the cavity of the macrocycle in a manner analogous to cation binding by "crown ethers" and "cryptands", and that coordinatively saturated tin atoms, which can become pentacoordinate, are good candidates for the Lewis acids. Selective binding properties that correlate to the fit of an anion or donor in the organized binding region of the macromolecular host were expected, and such properties might be exploited for anion separation, anion identification, and catalysis.

A series of macrobicyclic hosts containing two Lewis acidic tin atoms as binding sites was developed, and anion binding was studied by  $^{119}\text{Sn}$  NMR spectroscopy (TR-1, Pub-2). Tricyclic, ditopic hosts containing four Lewis acidic tin atoms were developed (TR-2, Pub-3). X-ray crystal structure determinations of a number of the hosts were obtained (TR-4, TR-5, TR-6, TR-7, TR-8, TR-9, TR-10, TR-11, Pub-5). Solid complexes of macrobicyclic hosts binding anions were isolated and studied by X-ray crystallography and solid state  $^{119}\text{Sn}$  NMR spectroscopy (Pub-1). Force field parameters for compounds containing tetrahedral tin atoms were developed and evaluated (TR-3, TR-12, Pub-4). The structures of the macrobicyclic hosts were studied by molecular mechanics calculations, and the results were compared to the crystal structures (TR-13, Pub-5).



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## List of Technical Reports

1. "Macrocycles Containing Tin. The Preparation of Macrobicyclic Lewis Acidic Hosts Containing Two Tin Atoms and  $^{119}\text{Sn}$  NMR Studies of Their Chloride and Bromide Binding Properties in Solution", Michael T. Blanda, John H. Horner, Martin Newcomb, May 24, 1989
2. "Macrocycles Containing Tin. Ditopic, Tricyclic. Lewis Acidic Hosts with Four Binding Sites", Michael T. Blanda and Martin Newcomb, August 30, 1989
3. "A Preliminary Set of Parameters for Force Field Calculations of Compounds Containing Tetrahedral Tin", John H. Horner and Martin Newcomb\*, May 24, 1990
4. "X-Ray Crystal Structure of 1,1,6,6,11,11,16,16-Octaphenyl-1,6,11,16-tetrastannaeicosane", Martin Newcomb and John H. Horner, November 7, 1990
5. "X-Ray Crystal Structure of 1,7-Diphenyl-1,7-distannabicyclo[5.5.5]heptadecane", Martin Newcomb and John H. Horner, November 16, 1990
6. "X-Ray Crystal Structure of 1,10-Diphenyl-1,10-distannabicyclo[8.8.8]hexacosane", Martin Newcomb, John H. Horner and Joseph P. Riebenspies, November 21, 1990
7. "X-Ray Crystal Structure of 1,12-Diphenyl-1,12-distannabicyclo[10.10.10]dotriacontane", Martin Newcomb and John H. Horner, November 26, 1990
8. "X-Ray Crystal Structure of 1,7-Dichloro-1,7-distannabicyclo[5.5.5]heptadecane", Martin Newcomb, John H. Horner, Michael T. Blanda, Chau Ha and Phillip J. Squatrito, November 28, 1990
9. "X-Ray Crystal Structure of 1,9-Dichloro-1,9-distannabicyclo[7.7.7]tricosane", Martin Newcomb, John H. Horner, Michael T. Blanda and Nancy McQuire, November 29, 1990
10. "X-Ray Crystal Structure of 1,11-Dichloro-1,11-distannabicyclo[9.9.9]nonacosane", Martin Newcomb and John H. Horner, November 30, 1990
11. "X-Ray Crystal Structure of 1,12-Dichloro-1,12-distannabicyclo[10.10.10]dotriacontane", Martin Newcomb and John H. Horner, December 3, 1990
12. "Parameters for Force Field Calculations of Compounds Containing Tetrahedral Tin", Martin Newcomb and John H. Horner, December 10, 1990
13. "Macrocycles Containing Tin. Crystal Structures and Molecular Mechanics, Calculated Structures of Macrobicyclic Lewis Acidic Hosts", Martin Newcomb and John H. Horner, December 12, 1990

List of Publications:

1. "Macrocycles Containing Tin. Solid Complexes of Anions Encrypted in Macrobicyclic Lewis Acid Hosts", M. Newcomb, J. H. Horner, M. T. Blanda, P. J. Squattrito, *J. Am. Chem. Soc.* **111**, 6294-6301 (1989).
2. "Macrocycles Containing Tin. The Preparation of Macrobicyclic Lewis Acidic Hosts Containing Two Tin Atoms and  $^{119}\text{Sn}$  NMR Studies of Their Chloride and Bromide Binding Properties in Solution", M. T. Blanda, J. H. Horner, M. Newcomb, *J. Org. Chem.* **54**, 4626-4636 (1989).
3. "Macrocycles Containing Tin. Lewis Acidic Hosts with Four Binding Sites", M. T. Blanda, M. Newcomb, *Tetrahedron Lett.* **30**, 3501-3504 (1989).
4. "Parameters for Force Field Calculations of Compounds Containing Tetrahedral Tin", J. H. Horner, M. Newcomb, *Organometallics* **10**, 1732-1740 (1991).
5. "Macrocycles Containing Tin. Crystal Structures and Molecular Mechanics Calculated Structures of Macrobicyclic Lewis Acidic Hosts", J. H. Horner, P. J. Squattrito, N. McGuire, J. P. Riebenspies, M. Newcomb, *Organometallics* **10**, 1741-1750 (1991).

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